Characterizing SWCNT Dispersion in Polymer Composites*

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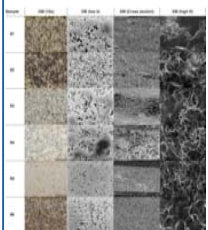
Abstract

The new wave of single wall carbon nanotube (SWCNT) infused composites will yield structurally sound multifunctional nanomaterials. The SWCNT network requires thorough dispersion within the polymer matrix in order to maximize the benefits of the nanomaterial. However, before any nanomaterials can be used in aerospace applications a means of quality assurance and quality control must be certified. Quality control certification requires a means of quantification, however, the measurement protocol mandates a method of "seeing" the dispersion first. We describe here the new tools that we have developed and implemented to first be able to "see" carbon nanotubes in polymers and second to measure or quantify the dispersion of the nanotubes.

Imaging the True Dispersion

Optical, electron and probe microscopy tools have been utilized in order to establish the effectiveness of visualizing carbon nanotubes in polymer matrices. However, the data extracted from these tools is insufficient to develop a quantifiable measure of the dispersion. What was needed was a measure of the 3-D distribution of the tubes. The new tools we developed allows for the collection of data from "Poly Transparent" imaging to begin to refine our models and understanding of the nature of the true dispersion.

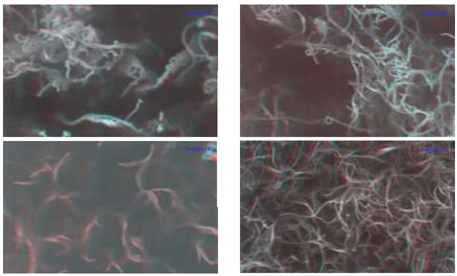


This figure shows the progression of some of the dispersion characterization tools and SWCNT dispersion procedures. Notice how the true nature of of the SWCNT dispersion becomes apparent under high kV Poly-Transparent imaging. The scale bar is 20μm 4μm, 1μn, and 500 nm for columns 1,2,3, and 4 repectively.

Sample 1: direct mixing: 1.0 vol.% LA-SWNT in CP2
Sample 2: *in-situ* polymerization: 1.0 vol.% LA-SWNT in CP2
Sample 3: *in-situ* polymerization under sonication: 1.0 vol.% LA-SWNT in CP2
Sample 4: *in-situ* polymerization under sonication: 0.5 vol.% LA-SWNT in CP2
Sample 5: *in-situ* polymerization under sonication: 0.5 vol.% LA-SWNT in β-CN
Sample 6: *in-situ* polymerization under sonication: 0.5 vol.% HiPCO-SWNT in β-CN

Poly-transparent 3-D Images

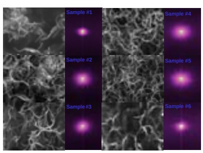
Poly-transparent imaging causes the non-conducting polymer to become transparent and allows the imaging of the conductive SWCNTs deep within the sample. Imaging the nanotubes in their natural state illustrates the effects that mixing conditions, sonication, polymer chemistry, and SWCNT composition have on the overall dispersion and resulting material properties. Poly-transparent imaging permits three-dimensional imaging of the SWNT network arrangement within the host polymer. The information derived from the three dimensional model provides the information necessary to determine a methodology to quantify the dispersion of the SWNT network within the host polymer.



A well dispersed sample, bottom right, is now easily distinguished from a poorly dispersed sample, upper left. The true progression of the 3-D SWCNT dispersion deep within the hosting polymer matrix can be visualized and related to the processing methods and resulting material properties.

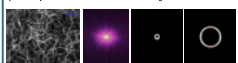
Image processing

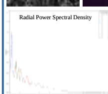
The dispersion of the nanotubes can be measured directly from the Poly-Transparent images by performing a series of image processing techniques. First we perform 2-D Fast Fourier Transform (FFT) analysis of the images.



RSPD plotting

Second we radially integrate over the spatial domain of the 2-D FFT to produce a plot of Radial Power Spectral Density (RSPD). The peaks of this plot correspond to dominate features within the image.





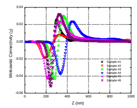
The RSPD measurements yield information relative of dominate features and ordering within the image. In this case, information associated with bundle diameter, segment length, and spacing within the nanotube network can be determined by analyzing the RSPD plot.

Fractal Dimension

The degree of ordering, or randomness, of the samples must also be determined to effectively measure the dispersion. The degree of randomness can be quantifiably characterized by determining the fractal parameter based upon the partitioning function associated with the Poly-Transparent images.

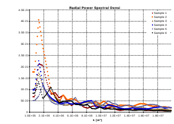
Minkowski Functions

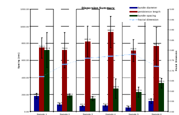
The Minkowski functional connectivity is another image analysis tool that can be utilized to verify the local structure characteristics gathered from RSPD plots and fractal dimension analysis.



Dispersion Summary

The dispersion characteristics of the six samples can be compared by observing the RSPD plots and the fractal dimension associated with each Poly-Transparent image.





The dispersion is expressed as an average bundle size, segment length, spacing between the bundles, and the fractal dimension. From this data we can quantitatively discern the effect that the parameters such as mixing conditions, sonication, polymer chemistry, and even the SWNT composition can have on the overall dispersion and resulting material properties of the composite samples..